

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

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<b>Date of mailing</b> (day/month/year) 03 November 2000 (03.11.00)	
<b>International application No.</b> PCT/GB00/00685	<b>Applicant's or agent's file reference</b> MRH/P15734WO
<b>International filing date</b> (day/month/year) 28 February 2000 (28.02.00)	<b>Priority date</b> (day/month/year) 26 February 1999 (26.02.99)
<b>Applicant</b> MILNER, Timothy, N. et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
 30 August 2000 (30.08.00)

☐ in a notice effecting later election filed with the International Bureau on:  
 \_\_\_\_\_

2. The election ☒ was

☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

<b>The International Bureau of WIPO</b> 34, chemin des Colombettes 1211 Geneva 20, Switzerland	<b>Authorized officer</b> Zakaria EL KHODARY
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

## PATENT COOPERATION TREATY

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## NOTIFICATION RELATING TO PRIORITY CLAIM

(PCT Rules 26bis.1 and 26bis.2 and  
Administrative Instructions, Sections 402 and 409)

From the INTERNATIONAL BUREAU

To:

HARRISON GODDARD FOOTE  
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ROYAUME-UNI

Date of mailing (day/month/year) 26 June 2000 (26.06.00)	<b>IMPORTANT NOTIFICATION</b>
Applicant's or agent's file reference MRH/P15734WO	
International application No. PCT/GB00/00685	International filing date (day/month/year) 28 February 2000 (28.02.00)
Applicant BRITISH NUCLEAR FUELS PLC et al	

The applicant is hereby notified of the following in respect of the priority claim(s) made in the international application.

1. ☒ **Correction of priority claim.** In accordance with the applicant's notice received on: 09 May 2000 (09.05.00), the following priority claim has been corrected to read as follows:  
US 04 March 1999 (04.03.99) 60/122,833  
☐ even though the indication of the number of the earlier application is missing.  
☐ even though the following indication in the priority claim is not the same as the corresponding indication appearing in the priority document:
2. ☐ **Addition of priority claim.** In accordance with the applicant's notice received on: , the following priority claim has been added:  
☐ even though the indication of the number of the earlier application is missing.  
☐ even though the following indication in the priority claim is not the same as the corresponding indication appearing in the priority document:
3. ☒ As a result of the correction and/or addition of (a) priority claim(s) under items 1 and/or 2, the (earliest) priority date is:  
26 February 1999 (26.02.99)
4. ☐ **Priority claim considered not to have been made.**  
☐ The applicant failed to respond to the Invitation under Rule 26bis.2(a) (Form PCT/IB/316) within the prescribed time limit.  
☐ The applicant's notice was received after the expiration of the prescribed time limit under Rule 26bis.1(a).  
☐ The applicant's notice failed to correct the priority claim so as to comply with the requirements of Rule 4.10.  
The applicant may, before the technical preparations for international publication have been completed and subject to the payment of a fee, request the International Bureau to publish, together with the international application, information concerning the priority claim. See Rule 26bis.2(c) and the PCT Applicant's Guide, Volume I, Annex B2(II).
5. ☒ In case where multiple priorities have been claimed, the above item(s) relate to the following priority claim(s):  
US 04 March 1999 (04.03.99) 60/122,833
6. A copy of this notification has been sent to the receiving Office and  
☒ to the International Searching Authority (where the international search report has not yet been issued).  
☒ the designated Offices (which have already been notified of the receipt of the record copy).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer  Yolaine CUSSAC
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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/GB00/00685 (22) International Filing Date: 28 February 2000 (28.02.00) (30) Priority Data: 60/121,854      26 February 1999 (26.02.99)      US 60/122,833      4 March 1999 (04.03.99)      US (71) Applicant (for all designated States except US): BRITISH NUCLEAR FUELS PLC [GB/GB]; Risley, Warrington, Cheshire WA3 6SAS (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): <u>MILNER</u> , Timothy, N. [GB/GB]; British Nuclear Fuels plc, Derwentwater Pavilion, Westlakes Science and Technology Park, Moor Row, Cumbria CA24 3JX (GB). <u>ROBBINS</u> , Rebecca, A. [GB/GB]; British Nuclear Fuel plc, Derwentwater Pavilion, Westlakes Science and Technology Park, Moor Row, Cumbria CA24 3JX (GB). <u>HOLMES</u> , Robert, G., G. [GB/GB]; British Nuclear Fuel plc, Derwentwater Pavilion, Westlakes Science and Technology Park, Moor Row, Cumbria CA24 3JX (GB). <u>SHEALY</u> , Stuart [US/US]; IT Corporation, Knoxville, TN (US). <u>MORREN</u> , Edward [US/US]; IT Corporation, Knoxville, TN (US). <u>AUSTIN</u> , Colin, R. [US/US]; MSC, Oakridge, TN (US).			(74) Agent: HARRISON GODDARD FOOTE; Tower House, Merriam Way, Leeds LS2 8PA (GB). (81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.
(54) Title: TREATMENT OF RADIOACTIVELY CONTAMINATED METALS			
(57) Abstract			
<p>Radioactively contaminated metal objects are treated with an acid solution to cause dissolution of a surface layer of the objects. The pH of the solution is then raised with calcium hydroxide and a magnesium containing compound to cause dissolved metal to separate from the solution in solid form. Further treatment steps may include addition of sodium hydroxide and nickel hexacyanoferrate, polymer assisted settling of solids, UV oxidation, ion exchange and filtration.</p>			

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## **TREATMENT OF RADIOACTIVELY CONTAMINATED METALS**

### **FIELD OF THE INVENTION**

This invention relates to the treatment of radioactively contaminated metals. For  
5 instance the present invention may be applied to the treatment of metal components  
which have been used in the nuclear power industry and have become radioactively  
contaminated. Such components can be cleaned using a combination of physical and  
chemical techniques following which they may be released onto the scrap metal  
market or melted and formed into plate or billets for recycling.

10

### **BACKGROUND OF THE INVENTION**

A chemical decontamination process utilises a series of immersion tanks to remove  
fixed radionuclides from the surface of metal components. Acid solutions contained  
in these immersion tanks dissolve scale, corrosion products and some base metal to  
15 effect removal of surface contamination. The various metal species and  
radionuclides accumulate in the immersion tanks and, as a result, performance  
deteriorates and the decontaminant must be changed.

### **STATEMENTS OF INVENTION**

20 The present invention provides a flexible, robust decontamination and waste  
treatment process enabling high throughput in a minimal cost operating regime. The  
process of the invention is capable of generating a significant volume of radioactive  
scrap metal (RSM), ranging from PWR Inconel components, which are contaminated  
with activation and fission products, to aluminium components contaminated with  
25 transuranics.

According to the present invention there is provided a method of treating a  
radioactively contaminated metal object comprising contacting the object with an  
acid solution to cause dissolution of a surface layer of the metal object, and raising  
30 the pH of the resultant solution with calcium hydroxide and a magnesium containing  
compound to cause dissolved metal to separate from the solution in solid form.

Preferably the acid is a mixture of nitric and hydrofluoric acids.

5 Preferably the calcium hydroxide and magnesium containing compound are provided in the form of the mineral Dolomite.

10 Preferably sodium hydroxide is additionally used to raise the pH of the resultant solution. More preferably the sodium hydroxide is added after the calcium hydroxide and until the pH is raised to from 9 to 11, more preferably to about 9.5.

15 Preferably caesium removal from the resultant solution is effected by addition of nickel hexacyanoferrate. It is preferred that the nickel hexacyanoferrate is prepared "fresh *in situ*", that is to say immediately or shortly before addition to the resultant solution. This avoids degradation of the nickel hexacyanoferrate which decomposes on storage.

Preferably, a polymer is then added to assist settling of solids. More preferably, the polymer is an anionic polymer.

20 Preferably the resultant solution is subjected to UV oxidation, more preferably by adding sodium hypochlorite and subjecting the solution to UV radiation.

25 Preferably the resultant solution is subjected to ion exchange using the material clinoptillolite, a natural zeolite, in order to remove caesium not already removed by the nickel hexacyanoferrate treatment.

Preferably the resultant solution is subjected to mechanical filtration prior to disposal of the supernatant liquid.

**DETAILED DESCRIPTION OF THE INVENTION**

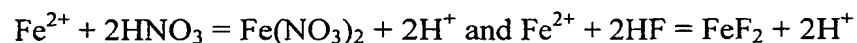
Figure 1 of the accompanying drawings is a flow diagram of a decontamination process in accordance with the present invention. As indicated in Figure 1, the process involves various unit operations. The incoming radioactive scrap (RS) is subjected to segregation and size reduction. It is then sentenced for physical chemical decontamination. Processing takes place through the decontamination line and a radionuclide contamination survey of the processed metals is carried out. The decontaminated metals are sent for recycling, disposal as LLW or sale into the scrap market. The spent decontamination liquor is treated and this results in solid/liquid separation. The process liquors are treated to meet the local sewer discharge limits. Process sludges are immobilised for disposal as non-RCRA dry active waste.

It is of importance in the context of the present invention that the chemical decontamination stage or stages provide a safe and cost effective process for the decontamination of RSM. The chemical decontamination should be capable of accepting a wide variation in feedstock and offer a cost saving over direct disposal of RSM. There should be produced a minimal volume of non RCRA secondary waste and the process liquors should be suitable for discharge to the local sewer.

Preferably the acid solution providing the decontamination of liquor comprises nitric and hydrofluoric acids which enable decontamination to be carried out on a varied or variable feedstock at high throughput levels. The aggressive nature of the nitric/hydrofluoric acid decontamination process requires careful control of the process parameters to achieve an optimum surface corrosion rate for a given substrate. Such optimising has various advantages. Base metal corrosion is kept to a minimum. The lifetime of the decontamination tank or tanks is prolonged. Uniform surface corrosion takes place without pitting of the base metal. The volumes of secondary waste are minimised by avoiding dissolution of uncontaminated base metal. There is also an avoidance of uncontrolled dissolution reactions which gives rise to off gases as the acids break down.

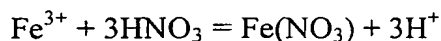
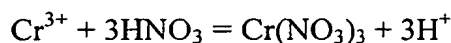
In order to achieve the optimal surface corrosion rate for a varied feedstock using a single chemical decontaminate, there is required a careful control of the temperature of the decontamination tank, the concentrations of the mixed acid treatments solution, the agitation and the oxidation potential of the decontaminant, the  
5 concentration of the dissolved metals and radionuclides in the decontamination tanks and the sequencing and immersion time in the decontamination tanks.

As fresh acid solution contacts a contaminated metal surface, dissolution occurs and the metal is dissolved in the acid solution. This results in metal loading of the acid  
10 bath. Although the reaction of the acids upon the metal surface is complex and involves a number of equilibrium reactions, the dissolution of iron by nitric acid can be simplified to the following reaction:-

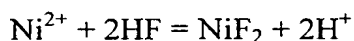
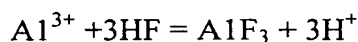


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Similar reactions take place with other metals such as:-



20



In the case of the dissolution of iron by nitric acid, there is a theoretical requirement for two moles of nitric acid to dissolve one mole of iron. However due to passivation  
25 of the metal surface, plate-out, oxidation of iron and the extremely slow dissolution rate as the acid is consumed in the dissolution reactions, such metal loading is impractical.

As a first order approximation, the metal dissolution rate of the acids is directly  
30 proportional to the concentration of the acids. Accordingly, for a high throughput of metal in the decontamination line, two factors have a significant impact on



decontamination performance. First, the overburdening of the acids with dissolved metals results in insufficient unreacted acid to achieve significant dissolution of the metal substrate. Secondly unreacted acid in the spent liquor has to be neutralised during waste treatment and this generates a significant volume of secondary waste.

5

Accordingly, the operation of the decontamination process of the present invention has involved a balance which has been struck between metal loading of the acids and dissolution rates, taking into account the potential for surface pitting and off gas production. It is important to minimise the production of off gasses that require scrubbing, thereby resulting in the generation of a secondary waste form and depletion of the acid capacity. This is particularly important when decontaminating the more reactive metals such as carbon steel and aluminium.

In order to obtain the optimisation mentioned above, it is preferred that the process is operated using a plurality of decontamination tanks in a manner referred to as "lag" basis. This procedure involves designating the decontamination tanks as low, medium and high dissolved metal content, each fulfilling a particular duty within the overall decontamination process. The low tank is used for final polishing of the decontaminated metal as this tank has the lowest radionuclide inventory and the highest dissolution kinetics. The high tank is used for initial decontamination where the highest levels of radionuclides, associated with the outermost surface, are removed. The medium tank is a transitional tank which is additionally used for "special" materials, which are substantially different from other materials of the feedstock. The use of the medium tank is optional.

25

During the process, the tanks are rotated from low to high based on a predetermined dissolved metal and radionuclide inventory. The high tank is taken off line and designated as spent once decontamination has become ineffective. In order to establish this there is carried out a regular sampling and analysis procedure to measure and record trends in tank performance. The procedure may be simplified with the cost of analysis reduced by using the experience gained from operating the

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process to devise a “finger print” which can be used to determine when a tank should be reclassified.

5 The operational flexibility of the chemical decontamination line allows both decontamination for recategorisation to LLW and also to free release. The economic case for free release or recategorisation has to be considered. In the case of a PWR primarily loop component which has been exposed to a high temperature, high pressure liquid environments generally require the removal of 20 to 30µm of corrosion film and base metal to achieve recategorisation to LLW. Achieving free  
10 release levels can require the removal of an additional 150 to 200µm of metal. Components from fuel cooling pools such as stainless fuel racks can be decontaminated to LLW by removing 5 to 8µm surface and free release can be achieved by removing 15µm of surface metal. The less demanding metal removal could favour free release.

15

The tank lag system allows decontamination to free release to be performed in the low activity tank and decontamination for recategorisation to LLW to be performed initially in the high and subsequently in the medium activity tank.

20 An additional advantage of the lag system is the degree of uniformity, in terms of dissolved metal and radionuclide content, that is introduced to the spent tank waste treatment process.

The spent acid resulting from the decontamination stage or stages may be subjected  
25 to a number of treatment steps. Treatment of the spent acids produces two waste streams. One is an alkaline wastewater which, after final polishing by chelant oxidation followed by ion exchange, is discharged to the local sewer. The other is a sludge containing the major metal species (iron, chromium, nickel) and radionuclides (cobalt, cesium) which may be directly solidified by grouting with, for instance,  
30 Portland cement to meet the appropriate waste acceptance criteria.

The spent acids from the chemical decontamination process contain a high concentration of dissolved metals and radionuclides, as illustrated in the following table:

Contaminant	Cr	Ni	Fe	Cu	$^{60}\text{Co}$	$^{134/137}\text{Cs}$	$^{241}\text{Am}$ $^{54}\text{Mn}$	Anions (nitrate fluoride chloride)
Concentration	1600 mg/L	2000 mg/L	12,500 mg/L	2000 mg/L	80,000 pCi/L	3000 pCi/L	1000 pCi/L	>10%

5

In treating the spent acids solution, the aim is to produce a minimal volume of non-RCRA solid waste. Since the composition of the metal component which is treated in the chemical decontamination stage affects the ratios of metals in the spent acid, a highly flexible approach is required. The waste treatment process may be either a batch process or a continuous process although reference will be made hereinafter to a batch process.

10

The waste treatment process may involve various steps as illustrated in Figure 2 of the accompanying drawings. As illustrated the spent acids solution is partially neutralised and conditioned using calcium hydroxide. The pH is then adjusted to a value in the range of from 9 to 10 using sodium hydroxide. Flocculation is then effected by seeding with inorganic adsorbents causing resultant coprecipitation of metals and radionuclides. Polymers are then added to aid gravity settling of solids. The solids and liquid are then separated and the solids are subjected to grouting. The wastewater liquid is then treated by means of UV/sodium hypochlorite to cause oxidation of chelate. Finally the waste water is polished by ion exchange using clinoptillolite as the resultant liquid is discharged to a local sewer.

15

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The above described process steps may be performed sequentially over a 24 hour period and the acid tank, when empty, is replenished with fresh acid and returned to service. The above described treatment process is both robust and simple to operate and uses readily available, low cost chemicals and process equipment.

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Calcium hydroxide is used principally to condition the metal hydroxide floc by assisting in the binding of leachable metals, principally chromium. Use of the magnesium containing compound improves the removal of hexavalent chromium. The calcium fluoride formed in the partial neutralisation forms the body of the floc and improves sludge settling characteristics. The calcium hydroxide is not used for pH control as this may result in the formation of an excessive amount of calcium sludge requiring disposal as a secondary waste product. Accordingly pH adjustment is carried out using sodium hydroxide, which is highly soluble and results in a sharp inflection point in pH, thereby allowing greater process control. The calcium hydroxide and the sodium hydroxide may be added together or in either order.

In the case where the acid includes hydrofluoric acid, the calcium from the calcium hydroxide forms insoluble calcium fluoride, making downstream processing of the supernatant liquid easier since the fluoride has been removed.

The presence of dissolved iron assists in chromium removal due to the  $\text{Fe (II)} \leftrightarrow \text{Fe (III)}$  couple ensuring the chromium is present in reduced Cr (III) state. In some cases, when the Fe (II)/Cr ratio in the spent acid is less than 4:1 wt/wt, additional ferrous iron may be required to reduce chromium to the less soluble form. This can also be controlled by management of the processing of nickel/chromium alloys and stainless or carbon steels in order to supply the appropriate iron ratio needed for chromium reduction.

The above described two-stage neutralisation results in the formation of a floc which contains virtually all the radionuclide and metal contaminants with the exception of cesium which is highly soluble. Typically cesium is removed from the wastewater by means of an ion exchange column. However such an approach has a number of disadvantages. The processing is slow due to the slow kinetics of the reactions. The highly selective ion exchange media which is required for column use in high salt streams is expensive. Less selective ion exchange media were rapidly exhausted by other metals and radionuclides, producing excessive secondary waste. Pretreatment

to remove particulate which has the potential to bind the columns is required and this produces an additional waste stream.

5 In a preferred method according to the present invention, cesium removal is affected using nickel hexacyanoferrate (HCF). A solid HCF ion exchanger is added in the amount of 300 ppm as a dilute slurry directly into the batch treatment tank once the pH has been adjusted. Caesium removal is aided by isotopic dilution by adding 10 ppm of non-radioactive caesium chloride. Preferably the HCF is used immediately after it has been formulated. Lower caesium removal and higher residual nickel  
10 resulted if the HCF is aged.

The kinetics of the cesium complexation with HCF is very rapid and the complex formed is removed from solution by adsorption and agglomeration with the metal hydroxide floc. A decontamination factor of 600 has been achieved for cesium and  
15 the supernatant liquor was free of all metals except a few ppm of nickel and chromium and trace levels of caesium. Final caesium removal was accomplished by adsorption on clinoptillolite, a natural zeolite.

Separation of the floc from the supernatant can be achieved by the use of mechanical  
20 separation devices. However such devices are either costly, generate a secondary waste in the form of a filter media or require frequent manual intervention. Accordingly it is preferred to make use of gravity settling and decanting. In order to speed the stages of the process, several hundred ppm of an anionic polymer was added to increase the rate of settling and the clarity of the supernatant. More  
25 preferably, the tank content are rotated using a stirrer device.

After a period of 4 to 8 hours the supernatant from the HCF step was decanted and processed through the waste water treatment polishing step. The solution was dosed with 0.25 to 0.5% sodium hypochlorite and treated with UV light to oxidise the  
30 soluble heavy metal complexes (nickel and cobalt-60). Insoluble compounds are produced. The treatment time was from 6 to 18 hours. The particulate is removed by

mechanical filtration by passing the material through a bed of sand and charcoal followed by cartridge filters.

As a result the concentration of heavy metals, primarily nickel, is reduced from 10 to 20 mg/L to the required discharge limit of not greater than 1.5 mg/L. The  $^{60}\text{Co}$  was similarly reduced from 20 to 50 pCi/L to the required discharge limit of less than 1 pCi/L.

If necessary, further caesium removal may be achieved by mixing the decant liquor with laundry water from the final effluent tank. This dilution lowered the concentration of nitrate and fluoride salts in the wastewater and improved the efficiency of the caesium removal by ion exchange using two columns packed with clinoptillolite. The clinoptillolite column had a residence time of 60 to 80 minutes and the resulting wastewater met local discharge criteria. The capacity of the ion exchange column was found to be between 80,000 to 90,000 bed volumes and the sand bed filter did not require to be changed out.

The second waste stream from the WTS comprises the radionuclide and metal contaminated sludge from the settling and decanting step. This sludge or metal floc was directly solidified in a cementitious grout. This was achieved by pumping the settled solids from the batch treatment tank into 55-gallon drums. A helical drum mixer was used to incorporate 0.75 to 1.0 pounds of portland cement per pound of sludge. This was accomplished with 20 to 30 minutes mixing and there was only a small heat rise during this process. The grout was then pumped from the drums into moulds that were designed to produce a final waste form meeting local acceptance criteria for size requirements for debris. The resulting grouted waste was produced with a minimal volume increase of 20%. It was cured overnight with no bleed and had a strength of over 50 psi.

Aspects of the present invention will now be described, by way of illustration only, in the following example.

A quantity of radioactively contaminated ferrous iron was loaded into a bath contain nitric and hydrofluoric acids and the acids were allowed to react with the surface of the iron until a desired metal loading in the bath had been achieved. Dolomitic lime  
5 (calcium hydroxide containing magnesium) was added to the bath in the form of a water slurry. The bath was then agitated for 30 minutes following which caustic soda (sodium hydroxide diluted with water) was added to give a pH in the bath of 9.5. The bath was agitated for a further 30 minutes following which caesium removal is effected by the addition of nickel hexacyanoferrate (HCF). The HCF was prepared  
10 "fresh *in situ*" to avoid degradation in storage, that is to say, it is added to the bath immediately after its preparation.

At this stage an anionic polymer (Nalclear) was added to the bath in order to promote the settling of the solids over a period of about 12 hours. The resultant solid sludge  
15 was separated from the supernatant and was immobilised in grout.

The supernatant was then treated with sodium hypochlorite which was added continuously while the bath was subjected to UV light radiation. At a pH of 9.5 solid nickel complexes are formed and removed by mechanical filtration. The pH was  
20 adjusted to 11.5 in order to remove solid cobalt complexes.

The resultant liquid may be treated by clinoptillolite ion exchange to remove further caesium, following which the liquor may be discharged to a sewer.

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## CLAIMS

1. A method of treating a radioactively contaminated metal object comprising contacting the object with an acid solution to cause dissolution of a surface layer of the metal object, and raising the pH of the resultant solution with calcium hydroxide and a magnesium containing compound to cause dissolved metal to separate from the solution in solid form.
2. A method according to Claim 1 wherein the acid is a mixture of nitric and hydrofluoric acids.
3. A method according to Claim 1 or Claim 2 wherein the calcium hydroxide and magnesium containing compound are in the form of Dolomite.
4. A method according to any of the preceding claims wherein the sodium hydroxide is additionally used to raise the pH of said resultant solution.
5. A method according to Claim 4 wherein the sodium hydroxide is added after the calcium hydroxide and until the pH is raised to from 9 to 11.
6. A method according to Claim 5 wherein the pH is raised to about 9.5.
7. A method according to any of the preceding claims wherein caesium removal from the resultant solution is effected by addition of nickel hexacyanoferrate.
8. A method according to Claim 7 wherein the residual hexacyanoferrate is prepared shortly before addition to the resultant solution.
9. A method according to any of the preceding claims wherein a polymer is added to the resultant solution to assist settling of solids.



10. A method according to claim 9 wherein the polymer is an anionic polymer.

11. A method according to any of the preceding claims wherein the resultant solution is subjected to UV oxidation.

5

12. A method according to Claim 11 wherein the UV oxidation is carried out by adding sodium hypochlorite and subjecting the solution to UV radiation.

10 13. A method according to any of the preceding claims wherein the resultant solution is subjected to ion exchange to effect caesium removal.

14. A method according to Claim 13 wherein the ion exchange is carried out using clinoptillolite.

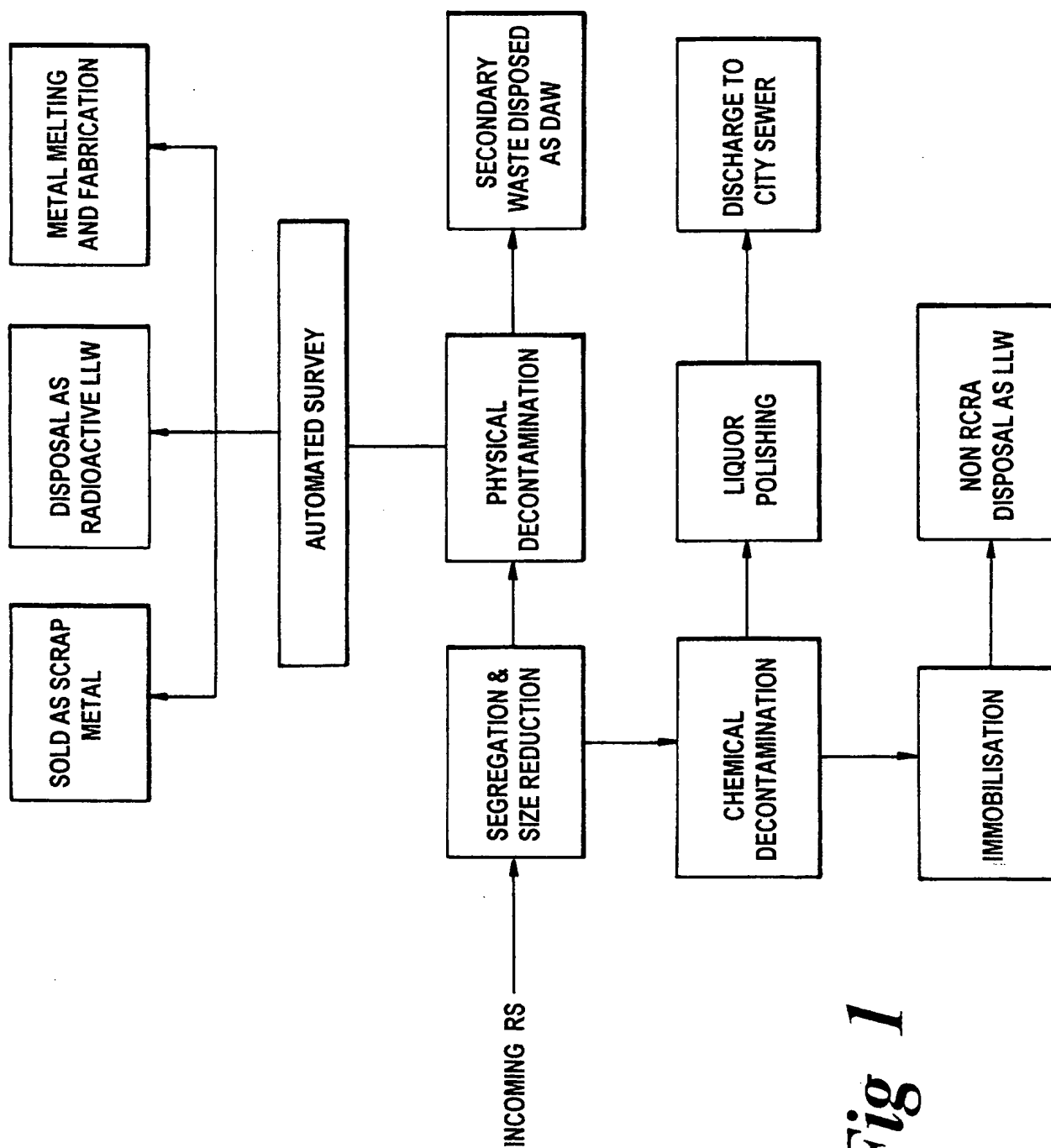
15 15. A method according to any of the preceding claims wherein the resultant solution is subjected to mechanical filtration prior to disposal of the supernatant liquid.

16 20 A method of treating a radioactively contaminated metal object comprising contacting the object with nitric and hydrofluoric acids to cause dissolution of a surface layer of the metal object, treating the resultant solution with calcium hydroxide and a magnesium containing compound followed by addition of nickel hexacyanoferrate and further subjecting the resultant solution to UV oxidation.

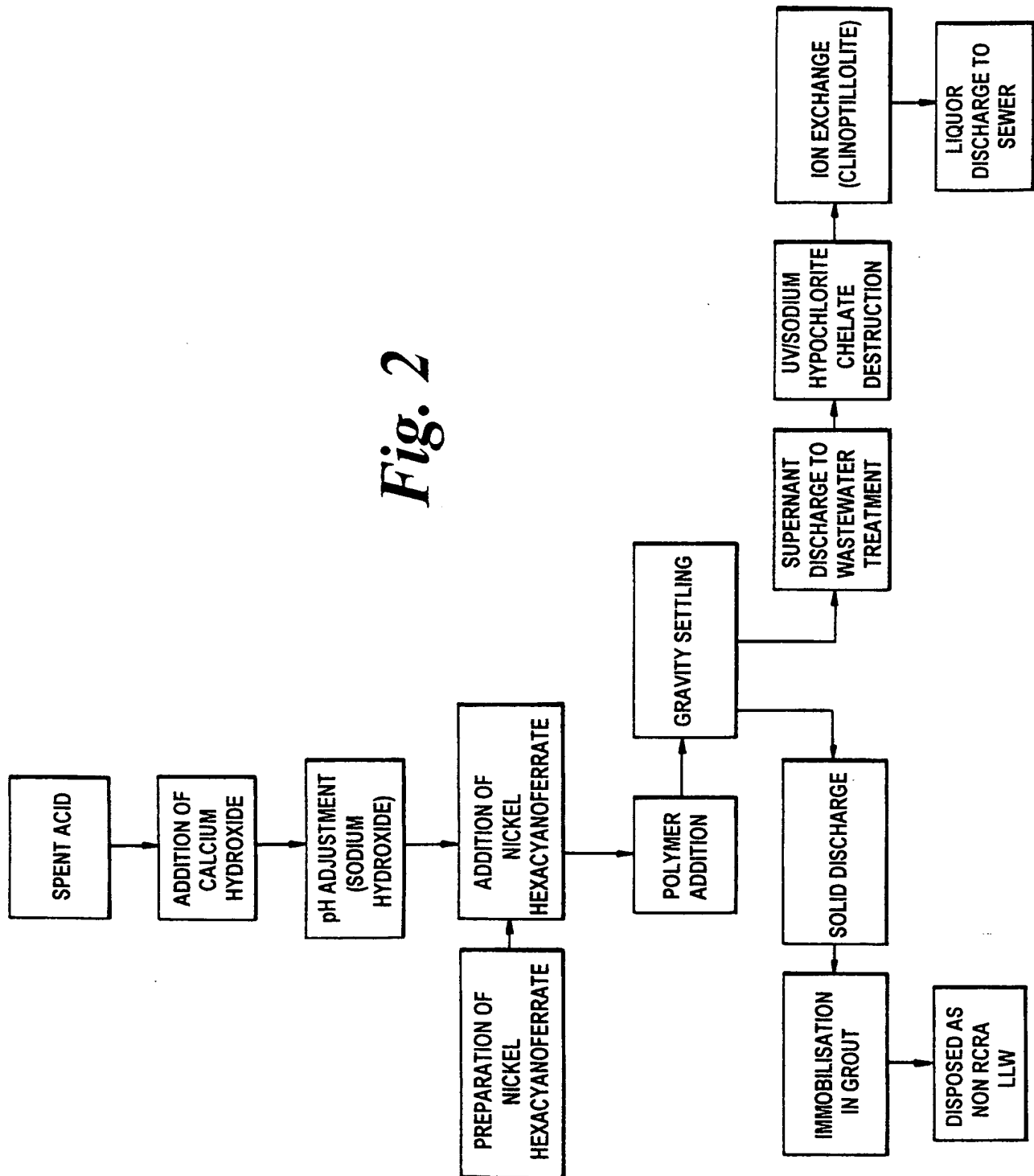
25

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1/2

*Fig 1*

2/2

*Fig. 2*

# INTERNATIONAL SEARCH REPORT

Inter:      nal Application No

PCT/GB 00/00685

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7      G21F9/00      C23G1/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7      G21F      C23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	FR 2 612 528 A (CHIMIDEROUIL) 23 September 1988 (1988-09-23) the whole document	1
A	HUSAIN A: "A PROCESS FOR DECONTAMINATING STAINLESS STEELS TO RELEASE LIMITS" NUCLEAR TECHNOLOGY, US, AMERICAN NUCLEAR SOCIETY. LA GRANGE PARK, ILLINOIS, vol. 85, no. 1, 1 April 1989 (1989-04-01), pages 66-73, XP000008933 ISSN: 0029-5450 the whole document	2

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

19 June 2000

Date of mailing of the international search report

29/06/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Brothier, J-A

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 00/00685

Patent document  
cited in search report

Publication  
date

Patent family  
member(s)

Publication  
date

FR 2612528

A

23-09-1988

NONE

# PATENT COOPERATION TREATY

From the:  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

Harrison Goddard Foote  
Tower House  
Merrion Way  
Leeds LS2 8PA  
GRANDE BRETAGNE

## PCT

### WRITTEN OPINION

(PCT Rule 66)

Date of mailing (day/month/year) <span style="float: right;">25.01.2001</span>	
Applicant's or agent's file reference <b>MRH/P15734WO</b>	<b>REPLY DUE</b> <span style="float: right;"><b>within 3 month(s)</b> from the above date of mailing</span>
International application No. <b>PCT/GB00/00685</b>	International filing date (day/month/year) <b>28/02/2000</b>
Priority date (day/month/year) <b>26/02/1999</b>	
International Patent Classification (IPC) or both national classification and IPC <b>G21F9/00</b>	
Applicant <b>BRITISH NUCLEAR FUELS PLC et al.</b>	

1. This written opinion is the **first** drawn up by this International Preliminary Examining Authority.
2. This opinion contains indications relating to the following items:
  - I ☒ Basis of the opinion
  - II ☐ Priority
  - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
  - IV ☐ Lack of unity of invention
  - V ☐ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
  - VI ☐ Certain document cited
  - VII ☐ Certain defects in the international application
  - VIII ☒ Certain observations on the international application
3. The applicant is hereby **invited to reply** to this opinion.
 

**When?** See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

**How?** By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

**Also:** For an additional opportunity to submit amendments, see Rule 66.4.  
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.  
For an informal communication with the examiner, see Rule 66.6.

**If no reply is filed,** the international preliminary examination report will be established on the basis of this opinion.
4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: **26/06/2001.**

Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer / Examiner <b>Randez Garcia, F</b> <hr/> Formalities officer (incl. extension of time limits) <b>Schuster-Kaechele, W</b> Telephone No. +49 89 2399 2281
---	---



**I. Basis of the opinion**

1. This opinion has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*):

**Description, pages:**

1-11 as originally filed

**Claims, No.:**

1-16 as originally filed

**Drawings, sheets:**

1/2-2/2 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

## WRITTEN OPINION

International application No. PCT/GB00/00685

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

### VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**



**Re Item VIII**


**Certain observations on the international application**

- 1). Claim 16 comprises all the features of claim 1 and is therefore not appropriately formulated as a claim dependent on the latter (Rule 6.4 PCT).
- 2). The units of weight, volume or pressure employed on page 10, lines 18-28, are not additionally expressed in terms of the units stipulated by Rule 10.1 (a) PCT.

## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference MRH/P15734WO	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB00/00685	International filing date (day/month/year) 28/02/2000	Priority date (day/month/year) 26/02/1999
International Patent Classification (IPC) or national classification and IPC G21F9/00		
Applicant BRITISH NUCLEAR FUELS PLC et al.		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 4 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 2 sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"><li>I <input checked="" type="checkbox"/> Basis of the report</li><li>II <input type="checkbox"/> Priority</li><li>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</li><li>IV <input type="checkbox"/> Lack of unity of invention</li><li>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</li><li>VI <input type="checkbox"/> Certain documents cited</li><li>VII <input type="checkbox"/> Certain defects in the international application</li><li>VIII <input type="checkbox"/> Certain observations on the international application</li></ul>		
Date of submission of the demand  30/08/2000	Date of completion of this report  11.05.2001	
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Randez Garcia, F  Telephone No. +49 89 2399 2234	



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/00685

## I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

### Description, pages:

1-9,11	as originally filed		
10	as received on	26/04/2001 with letter of	23/04/2001

### Claims, No.:

1-9	as originally filed		
10-16	as received on	26/04/2001 with letter of	23/04/2001

### Drawings, sheets:

1/2,2/2	as originally filed
---------	---------------------

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB00/00685

4. The amendments have resulted in the cancellation of:

- ☐ the description,      pages:
- ☐ the claims,      Nos.:
- ☐ the drawings,      sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes:	Claims	1-16
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-16
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-16
	No:	Claims	

2. Citations and explanations  
**see separate sheet**

**Re Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

- 1). None of the prior art documents considered disclose methods for treating radioactively contaminated metal objects by contacting the object with an acid solution and then raising the pH of the solution with calcium hydroxide and a magnesium compound as claimed in claim 1.
- 2). Thus, FR-A-2 612 528 discloses a method for treating metal objects with an acid solution which comprises neutralising the solution with calcium carbonate. The document is silent about applying it to radioactively contaminated metal objects and about using a magnesium compound.

- 3). Document

HUSAIN A: 'A PROCESS FOR DECONTAMINATING STAINLESS STEELS TO RELEASE LIMITS' NUCLEAR TECHNOLOGY, US, AMERICAN NUCLEAR SOCIETY. LA GRANGE PARK, ILLINOIS, vol. 85, no. 1, 1 April 1989 (1989-04-01), pages 66-73, XP000008933 ISSN: 0029-5450

discloses ion-exchange resins for regenerating acidic solutions employed for decontaminating stainless steels.

- 4). Hence, the subject-matter of claim 1 is novel and involves an inventive step.
- 5). Claims 2-16 are concerned with particular embodiments of the process according to claim 1. Therefore, they also meet the requirements of Article 33(2) and (3) PCT.

mechanical filtration by passing the material through a bed of sand and charcoal followed by cartridge filters.

As a result the concentration of heavy metals, primarily nickel, is reduced from 10 to 20 mg/L to the required discharge limit of not greater than 1.5 mg/L. The  $^{60}\text{Co}$  was similarly reduced from 20 to 50 pCi/L to the required discharge limit of less than 1 pCi/L.

If necessary, further caesium removal may be achieved by mixing the decant liquor with laundry water from the final effluent tank. This dilution lowered the concentration of nitrate and fluoride salts in the wastewater and improved the efficiency of the caesium removal by ion exchange using two columns packed with clinoptillolite. The clinoptillolite column had a residence time of 60 to 80 minutes and the resulting wastewater met local discharge criteria. The capacity of the ion exchange column was found to be between 80,000 to 90,000 bed volumes and the sand bed filter did not require to be changed out.

The second waste stream from the WTS comprises the radionuclide and metal contaminated sludge from the settling and decanting step. This sludge or metal floc was directly solidified in a cementitious grout. This was achieved by pumping the settled solids from the batch treatment tank into 250 litre (55-gallon) drums. A helical drum mixer was used to incorporate 0.75 to 1.0 kg of portland cement per kg of sludge. This was accomplished with 20 to 30 minutes mixing and there was only a small heat rise during this process. The grout was then pumped from the drums into moulds that were designed to produce a final waste form meeting local acceptance criteria for size requirements for debris. The resulting grouted waste was produced with a minimal volume increase of 20%. It was cured overnight with no bleed and had a strength of over 345 k Pa (50 psi).

Aspects of the present invention will now be described, by way of illustration only, in the following example.

10. A method according to claim 9 wherein the polymer is an anionic polym r.

11. A method according to any of the preceding claims wherein the resultant solution is subjected to UV oxidation.

5

12. A method according to Claim 11 wherein the UV oxidation is carried out by adding sodium hypochlorite and subjecting the solution to UV radiation.

13. A method according to any of the preceding claims wherein the resultant solution is subjected to ion exchange to effect caesium removal.

10

14. A method according to Claim 13 wherein the ion exchange is carried out using clinoptillolite.

15. A method according to any of the preceding claims wherein the resultant solution is subjected to mechanical filtration prior to disposal of the supernatant liquid.

15

16. A method according to Claim 1 wherein the acid solution comprises nitric and hydrofluoric acids and the treatment with calcium hydroxide and a magnesium containing compound is followed by addition of nickel hexacyanoferrate and by further subjecting the resultant solution to UV oxidation.

20

25

30 P15734.1

# PATENT COOPERATION TREATY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

## PCT

NOTIFICATION OF TRANSMITTAL OF  
THE INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT  
(PCT Rule 71.1)

To:

Harrison Goddard Foote  
Tower House  
Merrion Way  
Leeds LS2 8PA  
GRANDE BRETAGNE

15 MAY 2001 054498

Date of mailing  
(day/month/year)

11.05.2001

Applicant's or agent's file reference  
MRH/P15734WO

### IMPORTANT NOTIFICATION

International application No.  
PCT/GB00/00685

International filing date (day/month/year)  
28/02/2000

Priority date (day/month/year)  
26/02/1999

Applicant

BRITISH NUCLEAR FUELS PLC et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

#### 4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



European Patent Office  
D-80298 Munich  
Tel. +49 89 2399 - 0 Tx: 523656 epmu d  
Fax: +49 89 2399 - 4465

Authorized officer

Schuster-Kaechele, W

Tel. +49 89 2399-2281





# PATENT COOPERATION TREATY

# PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference <b>MRH/P15734WO</b>	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. <b>PCT/GB00/00685</b>	International filing date ( <i>day/month/year</i> ) <b>28/02/2000</b>	Priority date ( <i>day/month/year</i> ) <b>26/02/1999</b>
International Patent Classification (IPC) or national classification and IPC <b>G21F9/00</b>		
Applicant <b>BRITISH NUCLEAR FUELS PLC et al.</b>		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  <b>30/08/2000</b>	Date of completion of this report  <b>11.05.2001</b>
Name and mailing address of the international preliminary examining authority:  <div style="display: flex; align-items: center;"> <div>             European Patent Office              D-80298 Munich              Tel. +49 89 2399 - 0 Tx: 523656 epmu d              Fax: +49 89 2399 - 4465           </div> </div>	Authorized officer  <b>Randez Garcia, F</b>  Telephone No. +49 89 2399 2234



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/00685

## I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

### Description, pages:

1-9,11	as originally filed		
10	as received on	26/04/2001 with letter of	23/04/2001

### Claims, No.:

1-9	as originally filed		
10-16	as received on	26/04/2001 with letter of	23/04/2001

### Drawings, sheets:

1/2,2/2	as originally filed
---------	---------------------

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB00/00685

4. The amendments have resulted in the cancellation of:

- ☐ the description,      pages:
- ☐ the claims,      Nos.:
- ☐ the drawings,      sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes:	Claims	1-16
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-16
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-16
	No:	Claims	

2. Citations and explanations  
**see separate sheet**

**R Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

- 1). None of the prior art documents considered disclose methods for treating radioactively contaminated metal objects by contacting the object with an acid solution and then raising the pH of the solution with calcium hydroxide and a magnesium compound as claimed in claim 1.
- 2). Thus, FR-A-2 612 528 discloses a method for treating metal objects with an acid solution which comprises neutralising the solution with calcium carbonate. The document is silent about applying it to radioactively contaminated metal objects and about using a magnesium compound.

- 3). Document

HUSAIN A: 'A PROCESS FOR DECONTAMINATING STAINLESS STEELS TO RELEASE LIMITS' NUCLEAR TECHNOLOGY, US, AMERICAN NUCLEAR SOCIETY. LA GRANGE PARK, ILLINOIS, vol. 85, no. 1, 1 April 1989 (1989-04-01), pages 66-73, XP000008933 ISSN: 0029-5450

discloses ion-exchange resins for regenerating acidic solutions employed for decontaminating stainless steels.

- 4). Hence, the subject-matter of claim 1 is novel and involves an inventive step.
- 5). Claims 2-16 are concerned with particular embodiments of the process according to claim 1. Therefore, they also meet the requirements of Article 33(2) and (3) PCT.

## PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>MRH/P15734W0</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/GB 00/ 00685</b>	International filing date (day/month/year) <b>28/02/2000</b>	(Earliest) Priority Date (day/month/year) <b>26/02/1999</b>
Applicant  <b>BRITISH NUCLEAR FUELS PLC et al.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

## 1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of Invention is lacking** (see Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.

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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/00685

A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 7 G21F9/00 C23G1/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 G21F C23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	FR 2 612 528 A (CHIMIDEROUIL) 23 September 1988 (1988-09-23) the whole document	1
A	HUSAIN A: "A PROCESS FOR DECONTAMINATING STAINLESS STEELS TO RELEASE LIMITS" NUCLEAR TECHNOLOGY, US, AMERICAN NUCLEAR SOCIETY. LA GRANGE PARK, ILLINOIS, vol. 85, no. 1, 1 April 1989 (1989-04-01), pages 66-73, XP000008933 ISSN: 0029-5450 the whole document	2



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

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Date of the actual completion of the international search

19 June 2000

Date of mailing of the international search report

29/06/2000

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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